

EFEITOS DA APLICAÇÃO DE VINHAÇA NA FERTILIDADE DO SOLO

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1 RESUMO

O objetivo deste trabalho foi analisar os efeitos da aplicação de vinhaça sobre as características de fertilidade do solo. Um estudo de monitoramento desses parâmetros foi realizado em uma fazenda localizada no interior do estado de São Paulo, no período de 2012 a 2017. Informações mineralógicas e sobre a concentração de metais no solo foram reportadas. Alterações significativas nas propriedades químicas do solo ao longo do tempo foram observadas, sendo a aplicação da vinhaça um dos fatores que exerceram influência na variação das características do solo, em combinação com fatores ambientais e o manejo agrícola. A saturação por bases decresceu em média de 64 para 40% entre 2012 e 2017, respectivamente, enquanto o volume médio anual de vinhaça foi $188 \pm 31 \text{ m}^3 \text{ ha}^{-1}$. A tendência de crescimento ao longo dos anos do parâmetro saturação por alumínio foi evidente e os valores de pH foram em torno de 5. Apesar da adoção de aplicação da vinhaça nos solos proporcionar a disponibilidade de nutrientes e água, é recomendado que o monitoramento e controle da qualidade do solo seja sempre realizado, mantendo-se consequentemente, a sua fertilidade e a sustentabilidade da produção de cana-de-açúcar.

Palavras-chave: fertirrigação, dosagens de efluente, características químicas do solo, impactos ambientais.

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EFFECTS OF VINASSE APPLICATION ON SOIL FERTILITY

2 ABSTRACT

This study aimed to evaluate the effects of vinasse application on soil fertility characteristics. A monitoring study of soil fertility parameters was conducted on a farm fertigated with vinasse, located in the state of São Paulo, from 2012--2017. Mineralogical and soil metal concentration information was reported. Significant alterations in the chemical properties of the soil over time were observed, with vinasse application being one of the factors that influenced the variation in soil characteristics, combined with environmental factors and agricultural management. The base saturation decreased on average from 64 to 40% between 2012 and 2017, while the mean annual volume of vinasse was $188 \pm 31 \text{ m}^3 \text{ ha}^{-1}$. The increasing trend in the years for the parameter aluminum saturation was remarkable, and the

pH values were approximately 5. Although the application of vinasse to soils increases the availability of nutrients and water, monitoring and control of soil quality, fertility and the sustainability of sugarcane production are recommended at all times.

Keywords: fertigation, effluent dosage, soil's chemical characteristics, environmental impacts.

3 INTRODUCTION

In Brazil, sugarcane cultivation has played a significant socioeconomic role since colonial times. With the rise of ethanol as a renewable biofuel, Brazilian sugarcane production has become a global leader and has steadily increased due to improvements in plant varieties, crop management, and harvesting systems (KOHLHEPP, 2010). Sugarcane production in the 2020/21 harvest reached 654.8 million tons, yielding 41.25 million tons of sugar and 32.8 billion liters of ethanol. This resulted in 143 million tons of bagasse, 23 million tons of filter cake, 110 million tons of straw, 6.3 million tons of ash, and 426 billion liters of vinasse. For the 2021/22 harvest, there was a forecast decrease of 9.14% in ethanol production and 5.71% in sugar production compared with the previous harvest (CANA-DE-AÇÚCAR, 2021).

Vinasse is considered the main wastewater from the ethanol production process in sugar and alcohol plants. It is generated in large quantities and presents certain peculiar characteristics, such as high temperature; acidic pH; and high concentrations of organic matter, suspended solids (particles $> 1.2 \mu\text{m}$), calcium, potassium, nitrogen, and phosphorus, among other nutrients (FUESS; GARCIA, 2014). Owing to the significant negative impacts of directly discharging vinasse into water bodies, Ordinance No. 323 of 1978 prohibited its discharge into watercourses (BRASIL, 1978). Thus, the main, most viable, and economical destination for vinasse is its application to sugarcane crops

through a technique called fertigation (PRADO; CAIONE; CAMPOS, 2013). Over time, the standardization of vinasse disposal, involving environmental agencies, has been carried out through various ordinances and/or resolutions regulating the criteria and procedures for its application to soil. Currently in force in the state of São Paulo is Ordinance P4.231 of CETESB (Environmental Company of the State of São Paulo), entitled "Vinhaça – Criteria and procedures for the application of vinasse to agricultural soil," which is in its 3rd edition and 2nd version (CETESB, 2015).

Among the numerous advantages of applying vinasse, under technical criteria, in the soil, we can mention the increase in natural soil fertility and the consequent increase in agricultural productivity, pH, nutrient availability, and microbial activity that contributes to the increase in the soil aggregation state and water retention capacity (FUESS, 2013). In the study by Sivaloganathan et al. (2013), the control treatment presented agricultural productivity values for sugarcane and sugar of 78 t ha^{-1} and 9.02 t ha^{-1} , whereas the best results of fertigation with vinasse were achieved with the 1:10 dilution, with agricultural productivity values for sugarcane and sugar of 115.0 t ha^{-1} and 13.5 t ha^{-1} , respectively.

The controlled use of vinasse fertigation is also essential from an economic and environmental perspective because of the reduction in water withdrawal for irrigation and the use of synthetic chemical fertilizers. Oliveira et al. (2014) evaluated the effects of vinasse application (with or without mineral

fertilizers) on sugarcane cultivation, indicating better agricultural production when using only vinasse fertigation, both in terms of sugarcane productivity (49.98 t ha⁻¹) and sugar productivity (7.01 t ha⁻¹), thus reducing the use of mineral fertilizers.

However, inadequate application of vinasse to the soil can lead to the risk of contamination of the soil, groundwater and surface water due to the presence of ammonia and potentially toxic metals (mainly Al, Fe, Cu, Cr, Ni, Pb and Zn) in addition to causing changes in dissolved oxygen in water bodies (SOTO; BASSO; KIANG, 2017).

According to Fuess, Rodrigues and Garcia (2017), high levels of total dissolved solids (TDS>4000mg/L) and biodegradable organic matter in vinasse (>14 g/L) can favor salinization and increase soil acidity. These chemical changes can consequently also lead to physical changes such as soil density, total porosity and water storage and availability, implying decreasing or increasing variations in the behavior of soil hydraulic conductivity (SOTO; BASSO; KIANG, 2017; UYEDA et al., 2013).

Thus, when faced with scenarios that can be beneficial or harmful, the physical-chemical characteristics of vinasse and soil management with the appropriate and targeted application of fertilizers and/or amendments, in accordance with current national and state standards and legislation, have become important factors in sustainable effluent management (CAVALETT et al., 2012). It is also

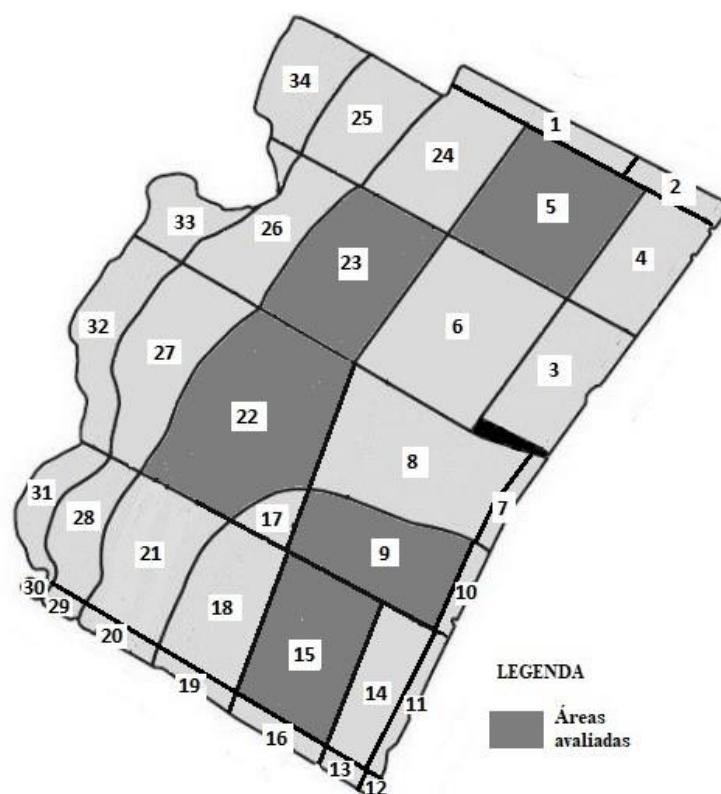
important to highlight the importance of prior knowledge about the chemical and mineralogical properties of the studied soil, since the types of clay minerals present in the soil are also factors that can influence physical-chemical changes, promote alterations in the leaching process of macro- and micronutrients, and facilitate the percolation of vinasse in the soil depth profiles (BUENO et al., 2009).

In this work, the physicochemical and mineralogical properties of the soil under vinasse application for sugarcane production were evaluated to identify the possible influences of vinasse dosage on soil fertility, with the goal of sustainable integration between effluent and agricultural management.

4 MATERIALS AND METHODS

4.1 Study area

The study area is located in the municipality of Pirassununga-SP, has an extension of 726.9 km² and an elevation of 627 m above sea level, and is situated at latitude 21°59'46" South and longitude 47°25'33" West. The sugarcane cultivation area, with approximately 553 ha, is subdivided into plots, which are presented in Figure 1 as numbers from 1--34. Data evaluations for the present study were carried out in plots 5, 9, 15, 22 and 23, and the period of application of vinasses in these plots covered the years 2012--2017.

Figure 1. Study area and its subdivisions into plots

The study area comprises three relief units: the Central Plateau of the Paraná Basin, the Eastern Plateau of the Paraná Basin, and the Peripheral Depression of São Paulo (IBGE, 2013), characterized by slopes ranging from gentle to hilly (FERREIRA; CAETANO-CHANG, 2008). The most predominant soil type is the Dystrophic Red--Yellow Latosol (LVAd), with a medium/clayey texture (smooth, undulating relief), indicating intense weathering of primary minerals, low cation exchange capacity, and a relative concentration of resistant clay minerals and/or iron and aluminum oxides and hydroxides. Generally, LVAd soils have good physical properties (high internal permeability, relative depth and porosity) and are mostly located in reliefs favorable for agricultural management. Its main limitation is the low availability of nutrients and toxicity due to exchangeable aluminum, which requires adequate

management with correctives and fertilizers (SANTOS et al., 2018).

The climate of the study area, according to the Köppen climate classification, is Cwa, a tropical highland climate with a rainy summer and a dry winter (RORIZ, 2014). Data from Lima (2016) indicate that the period of water deficit occurs during the dry season, from April to September, and the months with the highest precipitation occur from December to February. The irregular distribution of rainfall highlights the need for some types of irrigation systems after planting or harvesting, as these development stages are highly dependent on soil water. In this study, this variable is not explored, given the dense data package already presented in the Vinasse Application Plans (PAVs). However, it is worth reinforcing the need for rainfall monitoring in future studies.

4.2 Analysis of Vinasse Application Plans

In this study, the parameters analyzed for the physical-chemical

characterization of the soil and its fertility took into account the parameters recommended in CETESB Ordinance P4.231 (Table 1).

Table 1. Chemical parameters according to ordinance P4.231 (Environmental Company of the State of São Paulo, 2015)

Characterization of soil fertility	Environmental characterization of soil	
Exchangeable aluminum (Al)	Antimony (Sb)	Selenium (Se)
Calcium (Ca)	Arsenic (As)	Zinc (Zn)
Magnesium (Mg)	Barium (Ba)	VOC Scanning
Sodium (Na)	Cadmium (Cd)	SVOC Scan
Sulfate (SO ₂)	Lead (Pb)	
Potential acidity	Cobalt (Co)	
Potassium (K)	Copper (Cu)	
Organic matter (OM)	Chromium (Cr)	
CTC	Mercury (Hg)	
pH	Molybdenum (Mo)	

CEC cation exchange capacity; V% percentage of base saturation; VOC volatile organic compounds; SVOC semivolatile organic compounds.

Ordinance P4.231 establishes standards and procedures for the disposal of vinasse on the soil and requires sugar and alcohol mills to prepare and submit annually the Vinasse Application Plan

(PAV), which must contain the areas and dosage rates to be applied. The maximum vinasse dosage defined by the aforementioned ordinance is represented below (Equation 1) (CETESB, 2015):

$$m^3 \text{ de vinhaça} \cdot \text{ha}^{-1} = [(0,05 \cdot CTC_{\text{efetiva}} - ks) \cdot 3744 + 185] / kvi \quad (1)$$

where 0.05 corresponds to 5% of the CEC (cation exchange capacity, $\text{cmol}_c \text{ dm}^{-3}$); ks is the potassium concentration in the soil ($\text{cmol}_c \text{ dm}^{-3}$); 3744 is the constant for transforming the results expressed in $\text{cmol}_c \text{ dm}^{-3}$ to kg of potassium in a volume of 1 ha per 0.8 meters of depth; 185 is the mass, in kg, of K_2O extracted by the crop per ha, per cut; and kvi is the potassium concentration in the vinasse in kg of $\text{K}_2\text{O m}^{-3}$.

The data contained in the PAVs of the study area, derived from soil analyses of the plots for compliance with the ordinance, were compiled into spreadsheets that allowed for the analysis of the temporal evolution of soil chemical changes through graphs, enabling the evaluation of a

possible correlation between the use of vinasse in fertigation and soil fertility. The data collection for this research work therefore referred to the results of laboratory analyses of the PAVs during the period from 2012--2017, which were provided by CETESB in Mogi-Guaçu, SP.

4.3 Soil sampling and chemical analysis

Field sampling was conducted in 2018 at three locations within the study area. The selected areas and their coordinates are presented below: plot 3, longitude -47.493684 and latitude -21.944610; plot 23, longitude -47.498589 and latitude -21.937313; and plot 26, longitude -47.503523 and latitude -

21.937238. Notably, samples from plot 3 (control) were collected at a farm boundary where no vinasse was applied, allowing comparative evaluations of the analytical results related to mineralogy and metal concentration. However, the results regarding the fertility parameters of plot 3 were not reported in the PAVs.

At each point, soil samples were collected at depths of 30 and 100 cm to assess the distributions of minerals and heavy metals throughout the soil profile. A soil depth of 30 cm was chosen because nutrients and organic matter tend to concentrate in shallower soil layers, where ions are more mobile and the soil is disturbed (VIEIRA, 1996). According to Oliveira and Prado (1987), in oxisol-type soils, such as those in this study, a depth of 80 to 100 cm is ideal for assessing the leaching of metals present in the soil.

The soil samples were characterized in terms of mineralogy and metal concentration to assess whether the potential clay minerals present in the sampled soils would also be affected by vinasse application. The analyses were performed after the samples were weighed and sieved via X-ray fluorescence spectrometry (XRF), which was carried out at the X-ray Fluorescence Laboratory of the Department of Petrology and Metallogeny (DPM) of São Paulo State University "Júlio de Mesquita Filho" (Unesp), Rio Claro campus. The equipment used was a Philips X-ray fluorescence spectrometer model PW-2400, and the analytical procedures

were based on the methodology proposed by Nardy et al. (1997).

From the mineralogical analyses of the study area, the silica–alumina ratio ($\text{SiO}_2/\text{Al}_2\text{O}_3$) was obtained, represented by the following expression (Equation 2), which is considered an index of soil weathering:

$$K_i = \text{SiO}_2/\text{Al}_2\text{O}_3 \cdot 1,7 \quad (2)$$

where K_i is a measure of the proportions of kaolinite $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ and gibbsite $\text{Al}(\text{OH})_3$ (BAPTISTA; MADEIRA NETO; MENESES, 1998).

5 RESULTS AND DISCUSSION

The results were divided and discussed according to the characterization of the soil in terms of its chemical fertility (based on the data contained in the PAVs) and in terms of the environmental quality of the soil, taking into account its mineralogical properties and metal concentrations.

5.1 Assessments of chemical parameters for characterizing soil fertility

The data compiled from the PAVs between 2012 and 2017 regarding the chemical parameters for characterizing soil fertility in plots 5, 9, 15, 22 and 23 are presented in Tables 2 and 3.

Table 2. Chemical analyses of the soil samples from the studied plots

Year	Gleba	Ca ²⁺	Mg ²⁺	K ⁺	Al ³⁺	H+Al	SB	CTC
(mmolc dm ⁻³)								
2017	15	21.31	9.86	1.12	0.14	26	32.38	58.38
2017	22	12.51	5.54	1.58	0.84	36	19.69	55.69
2017	5	10.89	4.97	0.98	0.5	32	16.9	48.9
2016	5	5.82	2.09	0.72	0.59	17	8.64	25.64
2016	9	12.65	2.68	0.66	0.58	22	16.07	38.07
2015	9	14.3	6.21	2.39	0.16	17	22.92	39.92
2015	23	6.34	2.89	0.57	1.13	20	9.82	29.82
2015	5	8.76	4.56	0.35	0.5	18	13.68	31.68
2014	23	18.42	9.71	1.28	0.28	16	29.45	45,45
2014	5	12.26	7.39	1.26	0.59	16	20.92	36.92
2014	15	10.79	4.72	1.97	0.27	22	17.51	39.51
2013	22	14.36	5.8	0.52	0.04	17	20.71	37.7
2013	23	15.83	6.78	1.18	0.17	13	23.8	36.8
2013	5	17.21	6.76	0.95	0.21	10	24.94	34.94
2012	23	12.56	6	1.4	<0.02	13	19.97	32.97
2012	22	59.07	17.94	2.32	0.04	18	79.34	97.34
2012	15	12.54	4.9	1.9	0.12	17	19.36	36,36

Table 3. Chemical analyses of the soil samples from the studied plots

Year	Gleba	pH	K:Mg:Ca	V (%)	m (%)	MO (%)
2017	15	5.5	1:9:19	55.5	0.43	17
2017	22	4.9	1:3:8	35.4	4.09	18
2017	5	5.0	1:5:11	34.6	2.87	13
2016	5	4.8	1:3:8	33.7	6.39	8
2016	9	4.7	1:4:19	42.2	3.48	13
2015	9	5.3	1:3:6	57.4	0.69	13
2015	23	4.6	1:5:11	32.9	10.3	14
2015	5	5.0	1:13:26	43.2	3.53	12
2014	23	5.6	1:8:14	64.8	0.94	12
2014	5	5.4	1:6:10	56.7	2.74	10
2014	15	5.0	1:2:5	44.3	1.52	10
2013	22	5.2	1:11:28	54.9	0.2	11
2013	23	5.8	1:6:13	64.7	0.69	12
2013	5	5.5	1:7:18	71.4	0.82	12
2012	23	5.3	1:4:9	60.6	0	12
2012	22	5.7	1:8:25	81.5	0.05	33
2012	15	5.1	1:3:7	53.3	0.6	12

V% percentage of base saturation; m% percentage of aluminum saturation.

Table 4. Dosages of vinasse applied to the studied soils

Year	Gleba	Ks (cmol dm^{-3})	CTC (cmol dm^{-3})	Dosage ⁽¹⁾ ($\text{m}^3 \text{ha}^{-1}$)	%ks/CTC	K ⁺ (kg ha^{-1})
2017	5	0.10	4.89	242.00	2.04	76.62
2017	15	0.11	5.84	288.80	1.88	87.56
2017	22	0.16	5.57	209.55	2.87	123.52
2016	5	0.07	2.56	131.80	2.81	56.29
2016	9	0.07	3.81	216.86	1.73	51.60
2015	5	0.04	3.17	215.67	1.10	27.36
2015	9	0.24	3.99	12.50	5.99 ⁽²⁾	186.85
2015	23	0.06	2.98	176.61	1.91	44.56
2014	5	0.13	3.69	131.06	3.49	98.51
2014	15	0.20	3.95	62.35	4.99	154.02
2014	23	0.13	4.55	185.53	2.82	100.07
2013	5	0.10	3.49	161.13	2.72	74.27
2013	22	0.05	3.77	232.02	1.38	40.65
2013	23	0.12	3.68	144.03	3.21	92.25
2012	15	0.19	3.64	51.43	5.23 ⁽²⁾	148.54
2012	22	0.23	9.73	379.53	2.38	181.38
2012	23	0.14	3.30	92.68	4.25	109.45

Ks: soil potassium concentration; CEC: cation exchange capacity; K⁺ concentration potassium in vinasse;

(1) Calculation using the value of $k_{vi} = 3.00 \text{ (kg K}_2\text{O. m}^{-3}\text{)}$;

(2) Potassium concentration value in soil > 5% of CEC.

With respect to soil macronutrients, Vitti and Mazza (1998) reported that the ideal K:Mg:Ca ratio for improving sugarcane productivity is approximately 1:3:9 to 1:5:25. This optimized ratio, presented in Table 3, is met for all areas analyzed, except for plots 22 and 5 in 2013 and 2015, where magnesium concentrations were well above those recommended by the authors. Since potassium has a high leaching potential, it is important to consider the retention capacity of other cations (Mg and Ca) in the soil when planning the vinasse dosages to be applied to minimize the likely environmental impacts resulting from this agricultural management technique (SILVA; GRIEBELER; BORGES, 2006).

Table 4 presents the values of potassium concentrations and cation exchange capacity (CEC) and the relationship between these two variables, with the expected vinasse dosages computed according to Equation (1). Two

results exceeding those regulated by Ordinance P4.231 were obtained for the %ks/CEC ratio, indicating a soil potassium concentration (ks) greater than 5% of the CEC. The standard establishes that upon reaching this limit, vinasse application should be restricted to replacing this nutrient on the basis of the average extraction by the crop (185 kg of K₂O per hectare per cut).

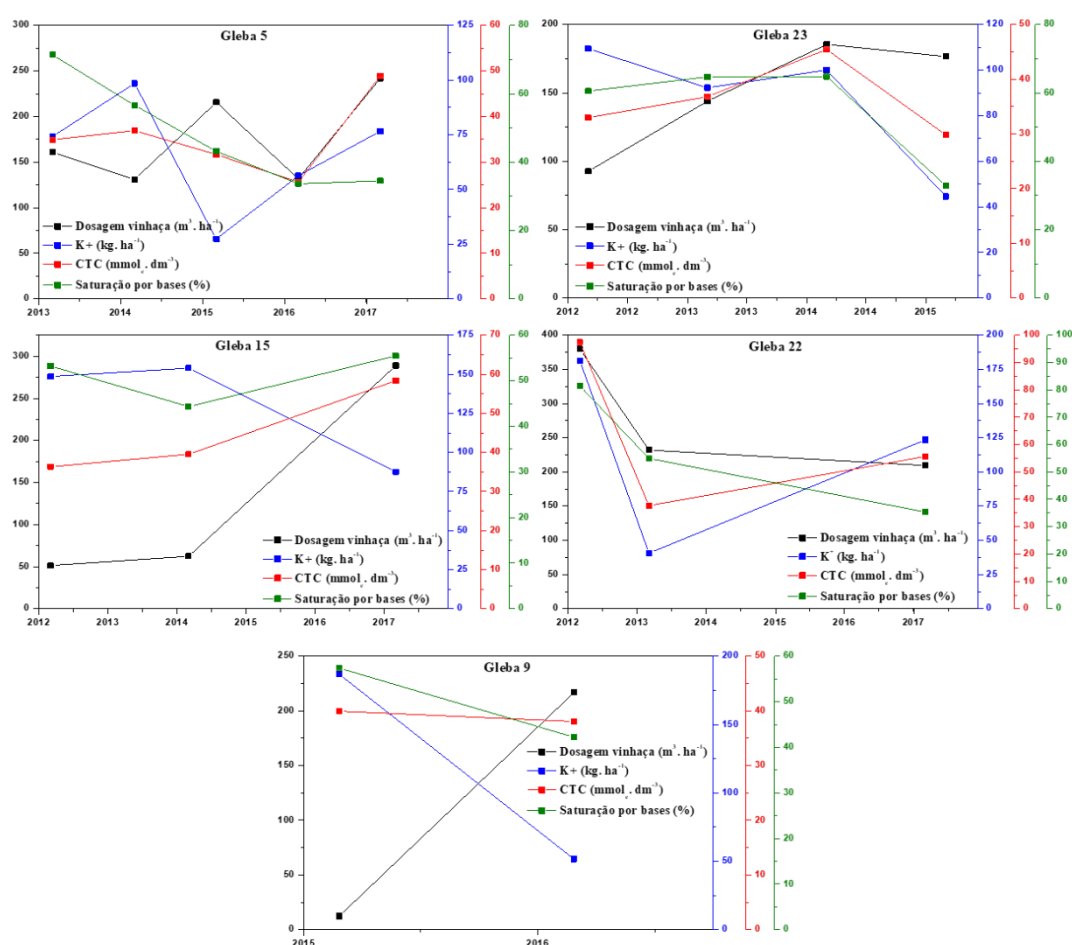
On the basis of the volumes of vinasse applied to the farm plots (Table 4), plot 5 presented dosages in five consecutive years (2013--2017), plot 23 presented dosage results in four consecutive years (2012--2015), plots 15 and 22 presented values for dosages in three years (2012, 2014 and 2017; 2012, 2013 and 2017, respectively), and plot 9 presented dosage results in two years (2015 and 2016), allowing the temporal analysis of the following chemical parameters in these plots: vinasse dosage, potassium concentration (K⁺), cation exchange

capacity (CEC), base saturation (V%), aluminum saturation (m%), potential acidity (H + Al) and pH. Importantly, the soil characterization obtained in one year was the result of the management carried out in the previous year.

Figure 2 shows the variations in the vinasse dosages applied to the soils of the five analyzed areas over time, as well as the corresponding potassium concentrations in the vinasse, the soil cation exchange

capacity, and the soil base saturation. The addition of vinasse promoted changes in all the analyzed parameters. The largest fluctuations in vinasse volume applied to the soil occurred in plot 5 from 2013--2017. For example, the dosages between 2014 and 2015 ranged from 125 to 225 $\text{m}^3 \text{ha}^{-1}$, respectively. The lowest vinasse dosages occurred in plot 15 between 2012 and 2014, with values above 50 $\text{m}^3 \text{ha}^{-1}$, and in plot 9 in 2015 (12.5 $\text{m}^3 \text{ha}^{-1}$).

Figure 2. Effect of vinasse dosage ($\text{m}^3 \text{ha}^{-1}$) on cation exchange capacity (CEC, $\text{mmol}_c \text{dm}^{-3}$) and base saturation (%) over the years in the studied areas.



Is expected to increase soil potassium concentrations, but other factors, such as leaching and nutrient uptake by the crop, can alter its final balance. The amount of potassium in the vinasse was 27.4 kg ha^{-1} in plot 5 and 186.9 kg ha^{-1} in plot 9 in 2015.

The soil potassium concentrations in plot 5 ranged from 0.35 to 0.72 $\text{mmol}_e \text{dm}^{-3}$ between 2015 and 2016 and from 2.39 to 0.66 $\text{mmol}_e \text{dm}^{-3}$ in plot 9 during the same period. The values found for the soil K⁺ concentrations are similar to the results of

Rossetto et al. (2004) when evaluating soils collected at the São José sugar mill (São Paulo state) at a depth of 0–25 cm. The K^+ concentrations found by the authors in the collected soils ranged from 1.0 to 2.20 $mmol_c dm^{-3}$ according to the treatment performed (addition of 50 to 200 kg $K_2O ha^{-1}$, or from 42 to 167 kg $K^+ ha^{-1}$). The data from this study indicate a significant response in sugarcane productivity to potassium application in most of the studied areas, with linear responses to the aforementioned relationship. On the other hand, potassium has a high leaching potential, depending on its presence at high concentrations, bioavailability in the soil, rainfall amount, and soil texture, among other factors (OTTO; VITTI; LUZ, 2010; ROSOLEM et al., 2006; TEJADA; GONZALEZ, 2006).

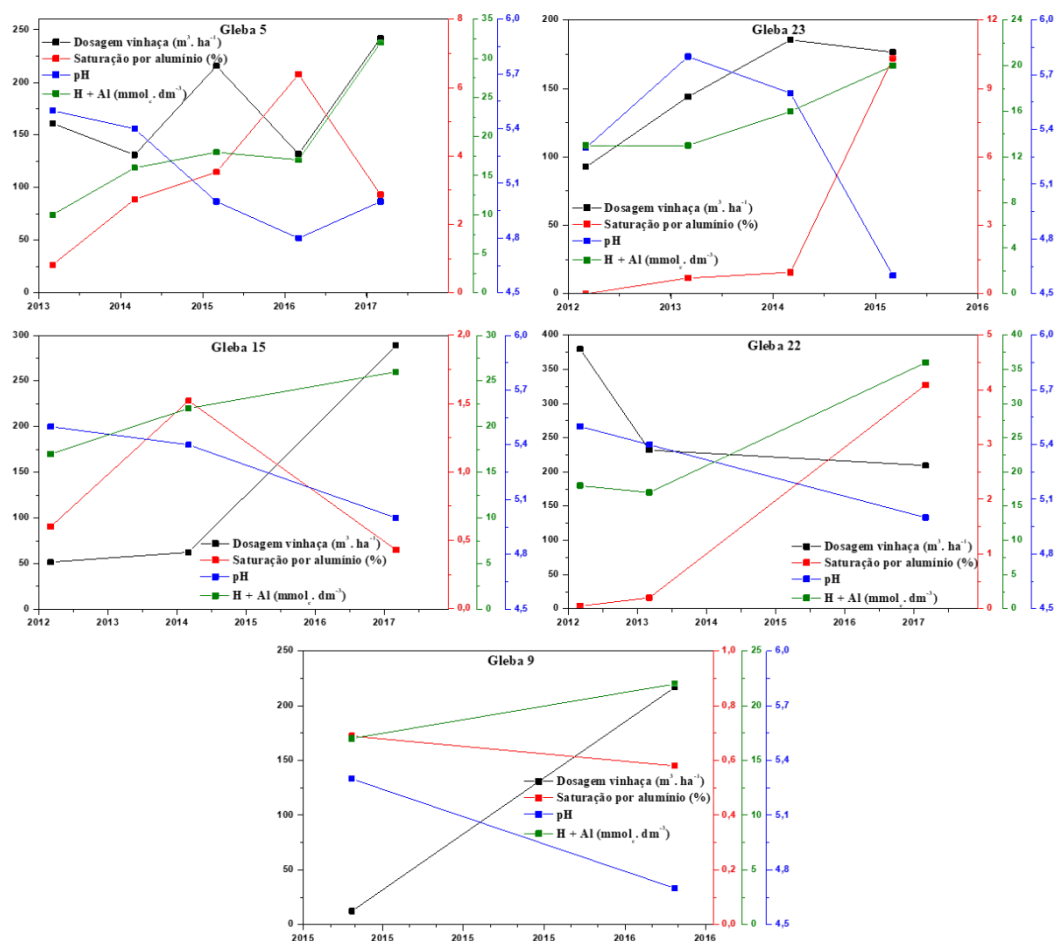
CEC has been identified as a good parameter for assessing K^+ availability in highly weathered soils (VAN RAIJ, 2011). A variation in CEC values is observed in these areas (Figure 2). Between 2012 and 2014, in plot 23, the number of vinasse applications increased from 90 to 175 $m^3 ha^{-1}$, and the CEC increased from 30 to 50 $mmol_c dm^{-3}$. The same effect of increased CEC due to vinasse application in a eutrophic Red–Yellow Ultisol was reported by Barros et al. (2010). The relationship between vinasse dosage and CEC may be related to the colloidal form of the organic matter present in the vinasse, which provides the soil with a greater amount of negative charges, attenuating the potential for cation leaching and regulating soil nutrient availability (GLÓRIA; ORLANDO FILHO, 1983). Importantly, no claim can be made about improved soil fertility due to increased CEC. This parameter is dependent on the effects of soil pH because it takes into account H^+ and Al^{+3} ions (RONQUIM, 2010), indicating that aluminum at levels toxic to plants and

adsorbed hydrogen ions that make a soil acidic may make up the predominant fraction of CEC.

According to Van Raij and Cantarella (1996), an ideal percentage for base saturation (V%), a parameter indicative of soil fertility, is 60% to promote an optimal balance for sugarcane crops. In annual average terms, base saturation (V%) decreased from 64 to 40% between 2012 and 2017, and vinasse dosages remained relatively constant between 2012 and 2016 (Table 3), resulting in an overall average of $176 \pm 13 m^3 ha^{-1}$ (or $188 \pm 31 m^3 ha^{-1}$, including 2017). Some authors have also reported a decrease in base saturation in the surface layer of soils cultivated with sugarcane after the application of vinasse and limestone (WATANABE; FIORETTO; HERMANN, 2004; VAN RAIJ et al., 1982).

The effects of vinasse dosage on pH, potential acidity ($H+Al$), and aluminum saturation in the soils of the five areas evaluated from 2012–2017 are shown in Figure 3. The vinasse dosages may have promoted significant changes in potential acidity, aluminum saturation, and pH. Between 2012 and 2017, the pH values varied between 4.6 and 5.8 (Table 3). According to Tomé Júnior's (1997) classification of soil acidity, the soils of the analyzed areas present high (pH 4.4 to 5) to low (pH 5.6 to 6) acidity. The reduction in pH values was more pronounced in plots 5 and 23, with values of 4.6 (pH 23 in 2016) and 4.8 (pH 5 in 2015), respectively. Some studies reported that no significant changes in pH were observed in areas fertigated with vinasse over the years and that the increase in pH due to vinasse dosages in the soils of these areas occurred mainly in areas where sugarcane cultivation has been carried out for at least 30 years (BEBÉ et al., 2009; SILVA; RIBEIRO, 1998).

Figure 3. Effect of vinasse dosage ($\text{m}^3 \text{ha}^{-1}$) on the variation in aluminum saturation (m%), pH and potential acidity ($\text{H} + \text{Al}$, $\text{mmol}_c \text{dm}^{-3}$) over time in the study area studied.



A decrease in pH and an increase in aluminum saturation can be observed (Figure 3). In plot 23, there was a clear increase in aluminum saturation between 2014 and 2015 (from 0.94 to 10.32) and a reduction in pH from 5.6 to 4.6. This scenario may be related to the successive increase in vinasse dosages applied in this area since 2012, where acidic vinasse contributed to the accelerated release of Al into the soil, since nutrient solubility is dependent on soil pH and the concentrations of metals such as Al^{3+} and Fe^{3+} can increase 1000-fold with a reduction of 1 pH unit (LINDSAY, 1979). Furthermore, soil weathering and the release of toxic Al can be more pronounced

under anaerobic conditions due to soil compaction, which contributes to significant increases or decreases in pH values (PRIMAVESI, 2006). Therefore, in addition to controlling the vinasse dosage applied to the soil, soil management is essential to prevent soil compaction during continuous cropping.

Osaki's (1991) guidelines indicate that aluminum saturation values above 20% begin to affect soil quality and are detrimental to agricultural crops. According to the literature, the high concentration of organic matter present in vinasse can increase the soil pH if biological stabilization is balanced. However, in aluminum-saturated soils, degradation is

disturbed, and the complexation of Al^{3+} with dissolved organic carbon is reduced, resulting in greater availability of this ion, which reacts with water, releases H^+ ions and acidifies the soil (CHRISTOFOLETTI et al., 2013).

The data resulting from the relationship between vinasse dosage and organic matter content were not plotted graphically, as the high concentration of organic matter present in vinasse is evident; therefore, both variables are positively correlated. Increasing the amount of soil organic matter through vinasse increases the cation retention capacity and, consequently, leaching losses. Another major advantage of vinasse fertigation is changes in soil physical conditions, increasing infiltration and water retention rates, contributing to aggregate formation and reducing soil susceptibility to erosion. High organic matter application increases the soil CEC and promotes the survival of

bacteria and fungi, which form humic acids and are responsible for the formation of macropores that facilitate air and water entry into the soil (SOUZA et al., 2015; RONQUIM, 2010).

5.2 Assessments of chemical parameters for the environmental characterization of soils

The mineralogical properties and metal contents of the soil were determined from soil samples collected from plots 3, 23 and 26 via X-ray fluorescence spectrometry (XRF) (Tables 5 and 6).

The total concentrations of metals, trace elements and rare earth elements were analyzed at two depths (30 and 100 cm) in soils from areas under fertigation treatment with vinasse (plots 23 and 26) and areas without vinasse application (plot 3) (Table 5).

Table 5. Total metal concentration (mg kg⁻¹) and guideline values in soil cultivated with sugarcane, fertigated with vinasse (plots 23 and 26) and without fertigation (plot 3), at two depths (30 and 100 cm).

	P1 30 cm	P1 100 cm	P2 30 cm	P2 100 cm	P3 30 cm	P3 100 cm	Guiding values		
	Plot 3		Plot 23		Plot 26		VP (2)	VRQ (2)	VI (2)
Barium ⁽¹⁾	28.0	44.5	53.9	38.0	305	65.8	120	75	500
Cobalt ⁽¹⁾	1.0	1.0	2.0	0.5	53.7	3.2	25	13	35
Copper ⁽¹⁾	7.1	6.8	8.9	8.3	123	28.5	60	35	760
Chrome ⁽¹⁾	80.7	92.0	83.9	172.3	176	91.1	75	40	150
Nickel ⁽¹⁾	3.5	5.4	1,2	7.0	69.6	23.2	30	13	190
Zinc ⁽¹⁾	14.7	15.1	9.0	3.8	106	36.8	86	60	1900
Cerium	0.7	1.0	1.0	1,2	28.9	66.6			
Strontium	3.4	1.0	2.0	6.9	43.7	2.0			
Gallium	16.5	18.9	17.7	15.8	17.7	19.4			
Yttrium	6.4	8.3	6.7	7.7	59.3	14.7			
Lanthanum	5.3	6.5	1.0	10.5	53.9	26.5			
Niobium	2.1	2.4	1.0	5.2	44.6	5.4			
Rubidium	7.0	6.0	5.6	6.1	15.6	10.9			
Vanadium	164	169	138	148	913	179			
Zirconium	201	189	186	198	306	213			

(1) Elements that appear in the list of ordinance P4.231 CETESB;

(2) Guiding Values for soil in the state of São Paulo from 11/22/2016 - CETESB: VP, prevention value; VRQ, quality reference value; VI, intervention value for agricultural soil.

Table 6. Mineralogical analysis of soil cultivated with sugarcane, fertigated with vinasse (plots 23 and 26) and without fertigation (plot 3), at two depths (30 and 100 cm).

Composition chemical	Chemical formula	Plot 3		Plot 23		Plot 26	
		P1 30 cm	P1 100 cm	P2 30 cm	P2 100 cm	P3 30 cm	P3 100 cm
Alumina	Al ₂ O ₃	7.41	7.00	5.51	5.79	19.64	10,12
Lime	Dog	0.03	0.05	0.04	0.04	0.34	0.09
Hematite	Fe ₂ O ₃	3.64	3.39	3.27	3.32	22.54	4.04
Phosphorus pentoxide	P ₂ O ₅	0.06	0.05	0.05	0.05	0.26	0.06
Pyrolusite	MnO	0.01	0.01	0.01	0.01	0.12	0.04
Magnesium oxide	MgO	0.02	0.01	0.01	0.01	0.22	0.05
Sodium oxide	On the 2 nd	0.01	0.01	0.01	0.02	0.02	0.02
Potassium oxide	K ₂ O	0.01	0.01	0.02	0.02	0.06	0.07
Rutile	TiO ₂	0.99	0.95	0.82	0.85	6.00	1.02
Silica	SiO ₂	83.44	84.60	87.00	86.68	31.12	79.33
Alumina	Al ₂ O ₃	7.41	7.00	5.51	5.79	19.64	10,12
Lime	Dog	0.03	0.05	0.04	0.04	0.34	0.09

The metals barium (Ba), cobalt (Co), copper (Cu), chromium (Cr), nickel

(Ni) and zinc (Zn) have guiding values according to Ordinance P4.231, defined as

the quality reference value (VRQ), which in turn is defined as the concentration of a certain substance in the soil that defines it as clean; prevention value (VP) is the concentration of a certain substance, above which harmful changes to soil quality may occur; and the intervention value (VI) is the concentration of a certain substance in the soil above which there are potential risks to human health (CETESB, 2020).

The control area (plot 3) presented high chromium concentrations (above the prevention values), which should be considered in the study of the surrounding areas (such as plots 23 and 26, which also presented concentrations above the VP), as they may be characteristics of the soil in question or evidence of contamination prior to the study. Vinasse commonly generated at high temperatures and low pH can contribute to the corrosion of storage tanks and pipes and cause effluent leakage, resulting in soil contamination (WILKIE; RIEDESEL; OWENS, 2000).

Metals such as Cd, Cr, Cu, Ni, Pb and Zn at concentrations of 2.4, 3.0, 15.7, 2.2, 8.8 and 14.1 mg/L (Table 5) were reported in vinasse samples from sugarcane molasses processing (CHANDRA et al., 2008). Notably, few studies have evaluated the occurrence and fate of metals in areas fertigated with vinasse (FUESS; RODRIGUES; GARCIA, 2017).

The concentrations of Ba, Cu, Cr, Ni, and Zn were above the prevention values for plot 26. The cobalt concentration would be above the intervention value, but caution should be taken when emphasizing this statement, as it may have resulted from contamination during sample collection, preparation, or analysis. The Cu concentrations analyzed in the present study were similar to those reported by Canellas et al. (2003), who reported significant increases in the Cu, Mn, and Zn contents in the profile of a silt-clayey Cambisol between depths of 20 and 40 cm. Ramalho and Amaral Sobrinho (2001) reported a

significant increase in P, Mn, and Co concentrations at depths of 0--10 cm in two types of soils studied: Cambisol and low-humic gneiss.

Plot 26 also presented high concentrations of the other elements. Vanadium and zirconium present the highest concentrations and may occur naturally, since in the study by Neves, Horn and Fraga (2008), the authors reported levels of Ba, Cr, Cu, Pb, V and Zn (226, 147, 175, 57, 403, 140 mg kg⁻¹, respectively) and the presence of lanthanum, cobalt and beryllium in a typical Red Yellow Latosol (LAVw).

Table 6 presents the results of the mineralogical characterization and chemical composition of the oxides present in the soil samples under study. High percentages of silica (SiO₂), ranging from 79.3 to 87%, were found for all areas and soil profiles surveyed, except for plot 26 at a depth of 30 cm (31.1%). The concentrations of alumina (Al₂O₃) and hematite (Fe₂O₃) presented maximum percentages of 19.6 and 22.5%, respectively, in addition to the relevant presence of rutile (TiO₂), which varied between 0.82% and 6.00%.

The large amounts of silica, iron and aluminum oxides indicate the presence of high amounts of silicates, possibly quartz, clay minerals, and secondary minerals derived from the weathering process (WU, 1981). These minerals, such as those observed in the study area, are characteristic of Red-Yellow Latosols such as those observed in the study area (NEVES; HORN; FRAGA, 2008). The heavy mineral rutile is also characteristic of the study area (ZANARDO et al., 2016).

In the three analyzed plots of the study area, the silica-alumina ratio (Table 7) was obtained according to Equation 2, indicating that the lower the *Ki* value is, the more weathered the soil is; that is, the silica has already been removed, with a subsequent increase in the alumina concentration. For these intensely

weathered soils, Ki is essentially a measure of the proportions of kaolinite $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ and gibbsite $\text{Al}(\text{OH})_3$

(BAPTISTA; MADEIRA NETO; MENESES, 1998).

Table 6. Silica–Alumina ratio in soil cultivated with sugarcane, fertigated with vinasse (plots 23 and 26) and without fertigation (plot 3), at two depths (30 and 100 cm).

Chemical composition	Formula	P1 30 cm	P1 100 cm	P2 30 cm	P2 100 cm	P3 30 cm	P3 100 cm
		Plot 3		Plot 23		Plot 26	
Alumina	Al_2O_3	7.41	7.00	5.51	5.79	19.64	10,12
Silica	SiO_2	83.44	84.60	87.00	86.68	31.12	79.33
K _i	$\text{SiO}_2 / \text{Al}_2\text{O}_3$.1.7	19:15	20.53	26.83	25.47	2.69	13.33

According to the silica–alumina ratio results, the sample from plot 26 at a depth of 30 cm had the lowest Ki index (2.69), indicating that the soil at this location may have undergone chemical weathering by hydrolysis, with partial elimination of silica and an increase in alumina, possibly resulting in runoff to surface waters. At a depth of 100 cm, the index was greater (13.33), indicating a higher silica concentration and less weathering due to this greater depth. For the other samples, the indices indicated a higher silica concentration and less weathering.

The instability and decomposition of minerals such as gibbsite and kaolinite can cause the release of Al ions into the soil, consequently increasing aluminum saturation and possibly soil acidity, which can be further aggravated by fertigation with vinasse (BAHIA et al., 2014). Therefore, determining mineral contents can also be an important tool for improving soil use and management.

The lowest concentrations of the other chemical compounds were, in order from lowest to highest percentages, Na_2O (0.01--0.02%), K_2O (0.01--0.07%), MnO (0.01--0.12%), MgO (0.01--0.22%) and CaO (0.03--0.34%). The similarity of the chemical analysis data corroborates the soil fertility parameters taken from the PAVs

(Table 2), indicating that an increasing percentage of the cations, i.e., $\text{CaO} > \text{MgO} > \text{K}_2\text{O}$, were present in the soil of the study area.

The samples from plot 3, considered the control area, presented the lowest K_2O contents (0.01%), indicating that the Red–Yellow Latosol of the study area has insufficient potassium and that the application of vinasse through fertigation replenishes this macronutrient as long as it is properly performed. In the areas where vinasse was applied (plots 23 and 26), the K_2O contents were relatively high, 0.02% for plot 23 and 0.06 and 0.07% for plot 26.

6 CONCLUSIONS

Technical monitoring of areas fertigated with vinasse is crucial for preventing soil degradation. This study presented a temporal analysis of some areas of a farm in the interior of São Paulo, where vinasse from an alcohol production plant was applied between 2012 and 2017. Increased soil acidity and aluminum concentrations were observed, as well as a decrease in base saturation (V%), indicating that soil fertility declined over a period of at least four years. Iron and aluminum oxides, which are part of the soil's colloidal fraction and, together with stabilized organic matter,

are capable of adsorbing cations, contributing to the increase in CEC, were identified in the soil. The concentrations of cobalt and chromium were higher than the intervention values, the causes of which, whether naturally occurring or due to anthropogenic influences, are unknown, indicating the need for further studies to understand this environmental phenomenon. It is possible that the application of vinasse in the volumes presented in this work per unit area is the cause of the relative soil degradation; however, other factors, such as precipitation, leaching, agricultural management and nutrient absorption by the

crop, are interdependent and should be taken into consideration in future studies to assess more assertively the aspects related to soil quality.

7 ACKNOWLEDGMENTS

The authors thank the São Paulo Research Foundation (FAPESP Grants: 2015/06246-7; 2016/24526-0 and 2017/18075-8) and the National Council for Scientific and Technological Development (CNPq Grant: 303469/2017-0) for their financial support.

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